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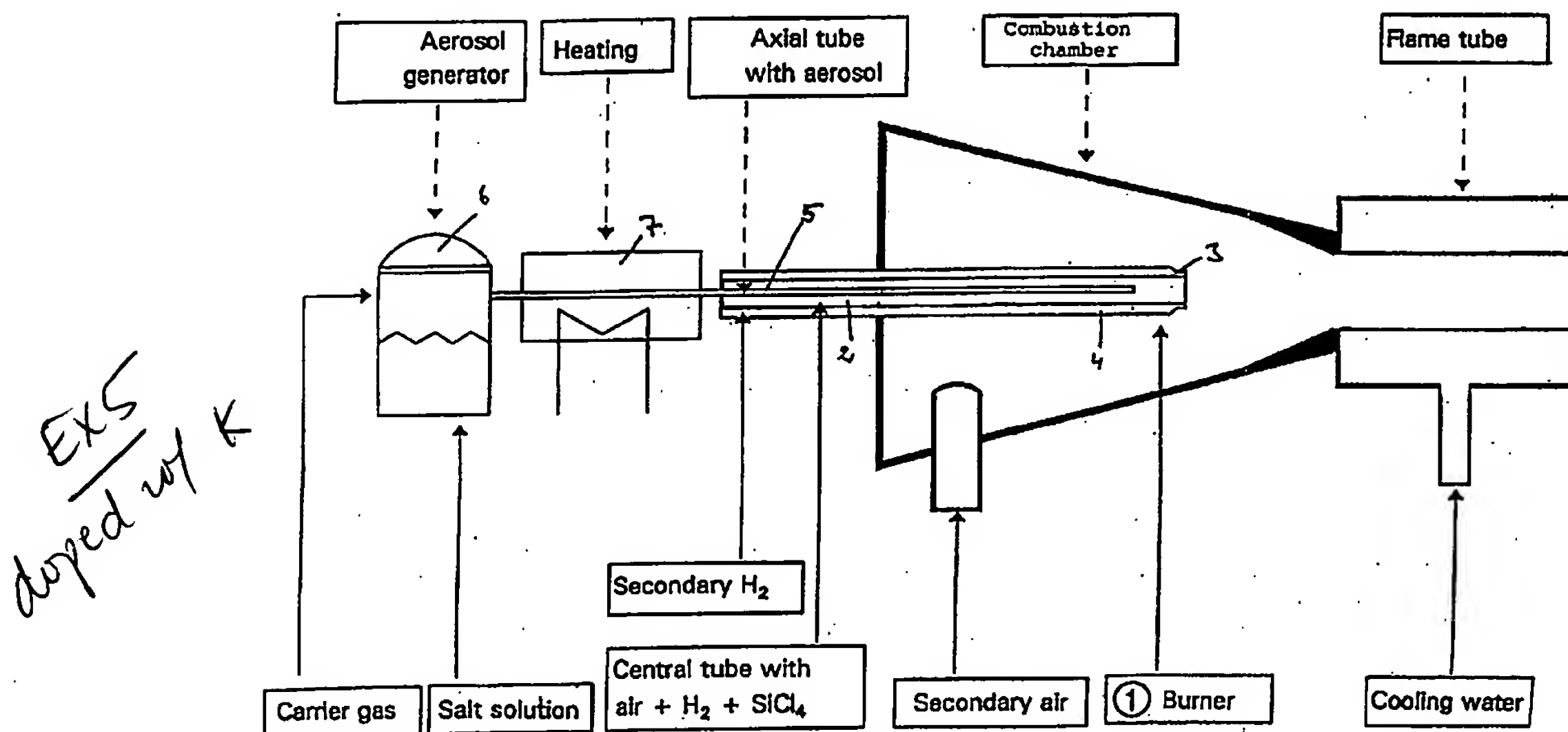
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(54) **OXYDES DOPES, PREPARES PAR VOIE PYROGENIQUE**

(54) **DOPED, PYROGENICALLY PREPARED OXIDES**

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breadth of the distribution
 $\frac{d_n}{d_a} = \text{arithmetic particle diameter}$
average particle diameter



(57) Oxydes de métaux et (ou) d'autres matériaux, préparés par voie pyrogénique, dopés avec un ou plusieurs constituants, à raison de 0,00001 à 20 % en poids. Le constituant de dopage peut être un métal et (ou) un autre matériau, ou un oxyde et (ou) un sel d'un métal et (ou) d'un autre matériau. L'aire surfacique BET de l'oxyde dopé peut se situer entre 5 et 600 m²/g. Les oxydes de métaux et (ou) d'autres matériaux, préparés par voie pyrogénique et dopés, sont préparés par addition d'un aérosol renfermant une solution aqueuse d'un métal et (ou) d'un autre matériau au mélange gazeux pendant l'hydrolyse à la flamme de composés vaporisables de métaux et (ou) d'autres matériaux. Les oxydes dopés préparés par voie pyrogénique peuvent servir, par exemple, comme charges.

(57) Doped, pyrogenically-prepared oxides of metals and/or non-metals, which are doped with one or more doping components in an amount of 0.00001 to 20 wt.%. The doping component may be a metal and/or a non-metal, or an oxide and/or a salt of a metal and/or a non-metal. The BET surface area of the doped oxide may be between 5 and 600 m²/g. The doped pyrogenically-prepared oxides of metals and/or non-metals are prepared by adding an aerosol, which contains an aqueous solution of a metal and/or non-metal, to the gas mixture during flame hydrolysis of vaporisable compounds of metals and/or non-metals. The doped pyrogenically-prepared oxides may be used, for example, as fillers.



Doped, Pyrogenically-Prepared Oxides

ABSTRACT

Doped, pyrogenically-prepared oxides of metals and/or non-metals, which are doped with one or more doping components in an amount of 0.00001 to 20 wt.%. The doping component may be a metal and/or a non-metal, or an oxide and/or a salt of a metal and/or a non-metal. The BET surface area of the doped oxide may be between 5 and 600 m²/g. The doped pyrogenically-prepared oxides of metals and/or non-metals are prepared by adding an aerosol, which contains an aqueous solution of a metal and/or non-metal, to the gas mixture during flame hydrolysis of vaporisable compounds of metals and/or non-metals. The doped pyrogenically-prepared oxides may be used, for example, as fillers.

Doped, Pyrogenically-Prepared Oxides

The invention relates to doped, pyrogenically-prepared oxides, a process for their preparation, and their use.

It is known that pyrogenically prepared oxides can be coated with metal salts or metal oxides by mixing the pyrogenically-prepared oxides with aqueous solutions of metal salts, and then drying and/or calcining.

10

Products prepared in this way have the disadvantages (a) that the doping substance is not homogeneously distributed in the entire primary particle, or (b) that, depending on the type of doping, inhomogeneities may occur during mixing. Thus, after doping and calcining, the primary particles of the doping substance may separate out and be present with much larger diameters than the primary particles of pyrogenic oxides.

- 20 It is an object of the invention to accomplish near homogeneous doping of pyrogenically-prepared oxides with another substance, while at the same time avoiding the presence of separate primary particles of the doping substance or oxides of the doping substance alongside primary particles of the pyrogenically-prepared oxide.

- The invention provides doped, pyrogenically-prepared oxides of metals and/or non-metals in which the basic components are pyrogenically-prepared oxides of metals and/or non-
- 30 metals, (using flame hydrolysis) which are doped with at least one doping component at 0.00001 to 20 wt.%, wherein the doping amount may preferably be in the range 1 to 10 000 ppm, and the doping component is a non-metal and/or

a metal or a non-metal salt and/or a metal salt or an oxide of a metal and/or a non-metal, and the BET surface area of the doped oxides is between 5 and 600 m²/g.

The invention also provides a process for preparing doped, pyrogenically-prepared oxides of metals and/or non-metals in which an aerosol is fed into a flame, (such as is used in a known manner to prepare pyrogenic oxides by flame hydrolysis), this aerosol being homogeneously mixed with
10 the gas mixture for flame oxidation or flame hydrolysis prior to reaction, and the aerosol/gas mixture is allowed to react in the flame and the resulting doped pyrogenically-prepared oxides are separated from the gas stream in a known manner. A salt solution or suspension which contains the components of the substance to be doped, which may be a metal salt or a non-metal salt (metalloid salt) or mixtures of both or a suspension of an insoluble metal compound or non-metal (metalloid) compound, is used as the starting material for the aerosol, and the aerosol
20 is prepared by nebulisation using a two-fluid nozzle or using an aerosol generator, preferably by the ultrasonic method.

The basic components which may be used are the non-metals/metals aluminium, niobium, titanium, tungsten, zirconium, germanium, boron and/or silicon.

The doping components which are used may be metals and/or non-metals and their compounds, provided they are soluble
30 in or can be suspended in a liquid solution. In a preferred embodiment, compounds of transition metals and/or noble metals may be used.

By way of example, cerium and potassium salts may be used as doping components.

The flame hydrolysis process for preparing pyrogenic oxides is known, for example, from Ullmann's Encyclopedia of Industrial Chemistry, 4th ed., vol. 21, page 464.

Due to the fine distribution of the doping component in the aerosol, and also the high temperatures (1,000 to 2,400°C) during subsequent flame hydrolysis, (during which the doping components are possibly further reduced in size and/or melted), the doping medium is finely distributed in the gas phase during the initial stages of production of the pyrogenic oxide, so that homogeneous incorporation of the doping component in the pyrogenically-prepared oxide is possible.

Using the process according to the invention, it is possible to dope all known pyrogenically-prepared oxides (e.g. SiO_2 , TiO_2 , Al_2O_3 , B_2O_3 , ZrO_2 , GeO_2 , WO_3 , Nb_2O_5) with other metal or metal oxides or non metal or non metal (metalloid) oxides or mixtures thereof.

The process according to the invention has several advantages. The aggregate or agglomerate structure of the pyrogenic oxide can be influenced by the choice of doping components. Furthermore, the pH of the pyrogenic oxide can be affected.

Catalytically active substances (e.g. cerium or noble metals) which are used as doping components can be distributed almost homogeneously in the pyrogenically-prepared oxide.

Phase conversion of pyrogenically-prepared oxides, for example from rutile to anatase in pyrogenically-prepared titanium oxide, can be affected by doping.

Using the process according to the invention, combinations of properties of pyrogenically-prepared oxides which have hitherto not been available, or available only with great difficulty, i.e. for example in processes requiring several steps, can be achieved.

10

Pyrogenically-prepared oxides of metals and/or non-metals, doped according to the invention, can be used as fillers, as support materials, as catalytically active substances, as starting materials for preparing dispersions, as polishing materials for polishing metal or silicon wafers in the electrical industry, as ceramic substrates, in the electronics industry (CMP applications), in the cosmetics industry, as additives in the silicone and rubber industry, to adjust the rheology of liquid systems, for heat-resistant stabilisation purposes, in the lacquer industry, as a heat insulation material, etc.

20

The invention also provides a device for performing the process according to the invention, which is characterised by an additional tube for introducing the aerosol arranged, preferably axially, in a burner of the structure known for preparing pyrogenic oxides, wherein the tube terminates upstream of the burner nozzle.

30

In another embodiment of the invention, the aerosol may be introduced using an annular nozzle, which may be arranged at any angle, preferably at right angles, to the main gas stream.

Specific examples illustrating the efficacy of the invention are described below, and make reference to the accompanying drawings, in which:

Figure 1 is a schematic diagram of apparatus similar to that used in the preparation of pyrogenic oxides, for introducing the aerosol into a flame;

Figure 2 shows an EM photograph of pyrogenic silica prepared in accordance with Example 3 (no doping); and

Figure 3 shows an EM photograph of pyrogenic silica prepared in accordance with Example 4 (doped with cerium salt).

The aerosol may be introduced into a flame in a preferred embodiment of the invention by means of a device like the one shown in Figure 1. In this case, the pipes for gas and aerosol introduction may also be interchanged.

Examples

The burner arrangement used in examples 1 to 4 is shown schematically in Figure 1.

According to Figure 1, the main part of the apparatus is a burner 1 of known construction, such as is conventionally used for preparing pyrogenic oxides.

Burner 1 consists of a central tube 2 which discharges at nozzle 3, from which the main gas stream flows into the combustion chamber and is there burned off. The inner nozzle is surrounded by the further annular nozzle 4

(mantle nozzle), out of which flows mantle or secondary hydrogen to prevent caking.

According to the invention, axial tube 5 is located inside central tube 2 and terminates a few centimetres upstream of nozzle 3 for central tube 2. The aerosol is fed into axial tube 5, wherein the aerosol gas stream from axial tube 5 is homogeneously mixed with the gas stream from central tube 2 over the last section of central tube 2.

10

The aerosol is produced in aerosol generator 6 (ultrasonic nebuliser). An aqueous salt solution which contains the metal or non-metal as a salt in dissolved or dispersed/suspended form is used as the aerosol starting material. The aerosol produced by aerosol generator 6 is passed through the hot zone 7 by means of a carrier gas stream, whereupon the water evaporates and small, finely distributed salt crystals remain in the gas phase.

← aerosol

Example 1 (no doping)

4.44 kg/h of SiCl_4 are evaporated at about 130°C and introduced into the central tube of the burner. 3 Nm^3/h of primary hydrogen and 8.0 Nm^3/h of air are also fed to the central tube. The gas mixture flows out of the inner nozzle of the burner and burns in the combustion chamber and the water-cooled flame tube connected in series therewith. 0.5 Nm^3/h of mantle or secondary hydrogen are fed to the mantle nozzle which surrounds the central nozzle, in order to prevent caking of the nozzle. An additional 12 Nm^3/h of secondary air are fed to the combustion chamber.

The aerosol flows out of the axial tube into the central tube. The aerosol consists of water vapour which has been produced in an amount of 195 g/h by ultrasonic nebulisation of pure distilled water in the aerosol generator.

The nebulised water vapour is passed through a heated pipe with the assistance of a carrier gas of about 0.5 Nm^3/h of air, wherein the aerosol is converted into gas at a temperature of about 180°C .

At the mouth of the burner (nozzle 3), the temperature of the gas mixture (SiCl_4 / air / hydrogen, water vapour and water aerosol) is 150°C .

The reaction gases and the resulting pyrogenic silica are passed under suction through a cooling system, by applying a reduced pressure to the flame tube, and thus cooled to about 100 to 160°C . The solid is separated from the vent gas stream in a filter or a cyclone.

The silica is produced as a white, finely divided powder. In a further step, adhering residues of hydrochloric acid

are removed from the silica by treating it with water vapour-containing air at elevated temperature.

The BET surface area of the pyrogenic silica is 150 m²/g.

5

The production parameters are given in Table 1. Further analytical data relating to the pyrogenic silica obtained are given in Table 2.

10 Example 2: (Doping with cerium)

The same procedure is used as described in example 1: 4.44 kg/h of SiCl₄ are evaporated at about 130°C and introduced into the central tube of the burner. 3 Nm³/h of
15 primary hydrogen and 8.0 Nm³/h of air are also supplied to the central tube. The gas mixture flows out of the inner burner nozzle and burns in the combustion chamber and the water-cooled flame tube connected in series therewith. In the mantle nozzle which surrounds the central nozzle,
20 0.5 Nm³/h of mantle or secondary hydrogen are supplied in order to prevent caking. An additional 12 Nm³/h of secondary air are supplied to the combustion chamber.

The aerosol flows out of the axial tube into the central
25 tube. The aerosol is a cerium salt aerosol which is produced in an amount of 210 g/h by ultrasonic nebulisation of a 5 % aqueous cerium(III) chloride solution in the aerosol generator.

30 The cerium salt aerosol is passed through a heated pipe with the assistance of 0.5 Nm³/h of air as carrier gas, wherein the aerosol is converted into a gas and a salt crystal aerosol at temperatures around 180°C.

At the mouth of the burner, the temperature of the gas mixture (SiCl_4 / air / hydrogen, aerosol) is 180°C .

The reaction gases and the resulting pyrogenically-prepared silica, doped with cerium, are removed under suction via a cooling system by applying a reduced pressure and thus cooled to about 100 to 160°C . The solid is separated from the gas stream in a filter or cyclone.

The doped, pyrogenically-prepared silica is produced as a white, finely-divided powder. In a further step, adhering hydrochloric acid residues are removed from the silica by treatment with water vapour-containing air at elevated temperatures.

15

The BET surface area of the doped, pyrogenically-prepared silica is $143 \text{ m}^2/\text{g}$.

The production parameters are given in Table 1.

20 Further analytical data for the pyrogenic silica obtained are given in Table 2.

Example 3 (no doping)

25 4.44 kg/h of SiCl_4 are evaporated at about 130°C and transferred to the central tube in the burner. $3 \text{ Nm}^3/\text{h}$ of primary hydrogen and $8.7 \text{ Nm}^3/\text{h}$ of air are also fed through the central tube. The gas mixture flows out of the inner nozzle of the burner and burns in the combustion chamber and the water-cooled flame tube connected in series therewith. In the mantle nozzle which surrounds the central nozzle, $0.5 \text{ Nm}^3/\text{h}$ of mantle or secondary hydrogen are supplied in order to prevent caking. An additional $12 \text{ Nm}^3/\text{h}$ of secondary air are supplied to the combustion chamber.

The aerosol flows out of the axial tube into the central tube. The aerosol consists of water vapour which is produced in an amount of 210 g/h by ultrasonic nebulisation of pure distilled water in the aerosol generator.

5

The aerosol is passed through a heated pipe with the assistance of about 0.5 Nm³/h of air as carrier gas, wherein the aerosol is converted into a gas at temperatures around 180°C.

10

At the mouth of the burner, the temperature of the gas mixture (SiCl₄ / air / hydrogen, water vapour or water aerosol) is 180°C.

15 The reaction gases and the resulting pyrogenic silica are removed under suction via a cooling system by applying a reduced pressure and thus cooled to about 100 to 160°C. The solid is separated from the gas stream in a filter or cyclone.

20

The silica is produced as a white, finely divided powder. In a further step, adhering hydrochloric acid residues are removed from the silica by treatment with water vapour-containing air at elevated temperature.

25

The BET surface area of the pyrogenic silica is 215 m²/g.

The production parameters are given in Table 1. Further analytical data for the pyrogenic silica obtained are given
30 in Table 2.

Example 4: (Doping with cerium)

The same procedure is used as described in example 1:

4.44 kg/h of SiCl_4 are evaporated at about 130°C and introduced into the central tube of the burner. $3 \text{ Nm}^3/\text{h}$ of primary hydrogen and $8.7 \text{ Nm}^3/\text{h}$ of air are also supplied to the central tube. The gas mixture flows out of the inner burner nozzle and burns in the combustion chamber and the water-cooled flame tube connected in series therewith. In the mantle nozzle which surrounds the central nozzle, $0.5 \text{ Nm}^3/\text{h}$ of mantle or secondary hydrogen are supplied in order to prevent caking. An additional $12 \text{ Nm}^3/\text{h}$ of secondary air are supplied to the combustion chamber.

The aerosol flows out of the axial tube into the central tube. The aerosol is a cerium salt aerosol which has been produced in an amount of 205 g/h by ultrasonic nebulisation of a 5 % aqueous cerium(III) chloride solution in the aerosol generator.

The cerium salt aerosol is passed through a heated pipe with the assistance of $0.5 \text{ Nm}^3/\text{h}$ of air as carrier gas, wherein the aerosol is converted into a gas and a salt crystal aerosol at temperatures around 180°C .

At the mouth of the burner, the temperature of the gas mixture (SiCl_4 / air / hydrogen, aerosol) is 180°C .

The reaction gases and the resulting pyrogenically-prepared silica, doped with cerium, are removed under suction via a cooling system by applying a reduced pressure and thus cooled to about 100 to 160°C . The solid is separated from the gas stream in a filter or cyclone.

The doped, pyrogenic silica is produced as a white, finely divided powder. In a further step, adhering hydrochloric

acid residues are removed from the pyrogenic silica by treatment with water vapour-containing air at elevated temperatures.

- 5 The BET surface area of the doped, pyrogenic silica is 217 m²/g.

The production parameters are given in Table 1. Further analytical data for the pyrogenic silica obtained are given
10 in Table 2.

→ Example 5: (Doping with potassium salts)

EX The same procedure is used as described in example 1,
15 wherein a 0.5 % aqueous potassium chloride solution is used as salt solution.

4.44 kg/h of SiCl₄ are evaporated at about 130°C and introduced into the central tube of the burner. 3 Nm³/h of
20 primary hydrogen and 8.7 Nm³/h of air are also supplied to the central tube. The gas mixture flows out of the inner burner nozzle and burns in the combustion chamber and the water-cooled flame tube connected in series therewith.
In the mantle nozzle which surrounds the central nozzle,
25 0.5 Nm³/h of mantle or secondary hydrogen are supplied in order to prevent caking.

The aerosol flows out of the axial tube into the central tube. The aerosol is a potassium salt aerosol which has
30 been produced in an amount of 215 g/h by ultrasonic nebulisation of a 0.5 % aqueous potassium chloride solution in the aerosol generator.

The potassium salt aerosol is passed through a heated pipe
35 with the assistance of 0.5 Nm³/h of air as carrier gas,

wherein the aerosol is converted into a gas and a salt crystal aerosol at temperatures around 180°C.

At the mouth of the burner, the temperature of the gas
5 mixture (SiCl_4 / air / hydrogen, aerosol) is 180°C.

The reaction gases and the resulting pyrogenically-prepared silica, doped with potassium, are removed under suction via a cooling system by applying a reduced pressure and the
10 particle/gas stream is thus cooled to about 100 to 160°C. The solid is separated from the gas stream in a filter or cyclone.

The doped, pyrogenically-prepared silica is produced as a
15 white, finely-divided powder. In a further step, adhering hydrochloric acid residues are removed from the silica by treatment with water vapour-containing air at elevated temperatures.

20 The BET surface area of the doped, pyrogenically prepared silica is 199 m²/g.

The production parameters are given in Table 1.

Further analytical data for the pyrogenic silica obtained
25 are given in Table 2.

Table 1

Experimental conditions during the preparation of doped pyrogenic silicas

No.	SiCl ₄ kg/h	Prim. air Nm ³ /h	Sec. air Nm ³ /h	H ₂ core Nm ³ /h	H ₂ mantle Nm ³ /h	N ₂ mantle Nm ³ /h	Gas temp. °C	Salt soln.	Aerosol amount kg/h	Air aeros. Nm ³ /h	BET m ² /g
Doping with cerium salt and comparison example											
1	4.44	8.0	12	3	0.5	0.3	150	only H ₂ O	0.195	0.5	150
2	4.44	8.0	12	3	0.5	0.3	180	5%CeCl ₃	0.210	0.5	143
3	4.44	8.7	12	3	0.5	0.3	180	only H ₂ O	0.210	0.5	215
4	4.44	8.7	12	3	0.5	0.3	180	5%CeCl ₃	0.205	0.5	217
Doping with potassium salt and comparison example											
3	4.44	8.7	12	3	0.5	0.3	180	only H ₂ O	0.210	0.5	215
5	4.44	8.7	12	3	0.5	0.3	180	0.5%KCl	0.215	0.5	199

Notes: Prim. air = amount of air in central tube; sec. air = secondary air; H₂ core = hydrogen in central tube;
 Gas temp. = gas temperature at the nozzle in the central tube; Aerosol amount = mass flow of salt solution
 converted into aerosol form; Air aerosol = carrier gas (air) in the aerosol.

Table 2

Analytical data for samples obtained according to examples 1 to 5

No.	BET [m ² /g]	Ce wt. µg/g	K wt. µg/g	Cl conc. ppm	LOD wt. %	LOI wt. %	Cl ppm	Grindo meter µm	Sedi- vol. %	Efficiency	pH 48 sus.	Comp. b. d. g/l	Thick. Lupoda l [mPas]
1	150	--			0.19	1.29		18	0	697	3.98	27	1745
2	143	1860	<5		0.09	1.33		20	0	690	3.93	26	1990
3	215	84	<5	45	0.27	1.87	45	18	11	422	4.00	25	3390
4	217	2350	<5	112	0.22	2.23	112	40	50	548	3.67	29	3680
5	199		300	55	0.32	1.86	55	60	50	451	4.83	32	2575

Notes: Cerium content as Ce in µg/g (ppm); Potassium content as K in µg/g; LOD = loss on drying (2h at 105°C, based on DIN/ISO 787/II, ASTM D 280, JIS K 5101/21); LOI = loss on ignition (2 h at 1000°C, based on DIN 55921, ASTM D 1208, JIS K 5101/23, with ref. to substance dried for 2 h at 105°C); Grindometer = Grindometer value; Sedi-vol. = sediment volume; Efficiency = turbidity measurement: the method of efficiency determination (turbidity measurement) is described in Patent DE 44 00 170; the suspension prepared by the same method is used for determining sediment volume after standing for a further 5 minutes; Compacted bulk density based on DIN/ISO 787/IX, JIS K 5101/18 (not sieved). Thickening in polyester reference system: described in EP-A 0 015 315.

$$0.34 \text{ g} / 1.1 \text{ g} = 30.6$$

Figure 2 shows an EM photograph of pyrogenic silica prepared in accordance with Example 3 (no doping).

Figure 3 shows an EM photograph of pyrogenic silica
5 prepared in accordance with Example 4 (doped with cerium salt).

It can be seen that the aggregate and agglomerate structure is modified when doped with cerium salt. Larger cohesive
10 structures are produced with doping.

The analytical data for silica in accordance with example 4, as compared with that for silica in accordance with example 3, shows an increased sediment volume and a greatly
15 increased efficiency value. This also indicates enlargement of the aggregate or agglomerate structure.

Furthermore, using silica doped with cerium in accordance with the invention, a clear improvement in thickening
20 effect is produced in unsaturated polyester resins.

The embodiments of the invention in which an exclusive property or privilege is claimed are defined as follows:

1. Doped, pyrogenically-prepared oxides of metals and/or non-metals, in which the basic components are oxides of metals and/or non-metals prepared by flame hydrolysis, doped with at least one doping component at 0.00001 to 20 wt.%, and in which the doping component is a non-metal and/or metal or a salt or an oxide of a metal or non-metal, and the BET surface area of the doped oxides is between 5 and 600 m²/g.
2. Doped, pyrogenically-prepared oxides of metals and/or non-metals as defined in Claim 1, in which the doping amount is in the range of 1 to 10,000 ppm.
3. A process for preparing pyrogenically-prepared oxides of metals and/or non-metals as defined in Claim 1, wherein an aerosol is fed to a flame such as is used for preparing pyrogenic oxides by flame hydrolysis, the aerosol being homogeneously mixed with the gas mixture for flame oxidation or flame hydrolysis prior to reaction, the aerosol/gas mixture is allowed to react in the flame and the resulting doped pyrogenically-prepared oxides are separated from the gas stream, in which a salt solution or suspension which contains the components of the substance to be doped is used as the starting material for the aerosol, and the aerosol is produced by nebulisation.
4. A process as defined in Claim 3, in which the components of the substance to be doped include a metal salt or non-metal salt (metalloid salt) or mixtures of

both, or a suspension of an insoluble metal compound or non-metal (metalloid) compound or mixtures of both.

5. A process as defined in Claim 3 or 4, in which the aerosol is produced by nebulisation using a two-fluid nozzle.

claim 7

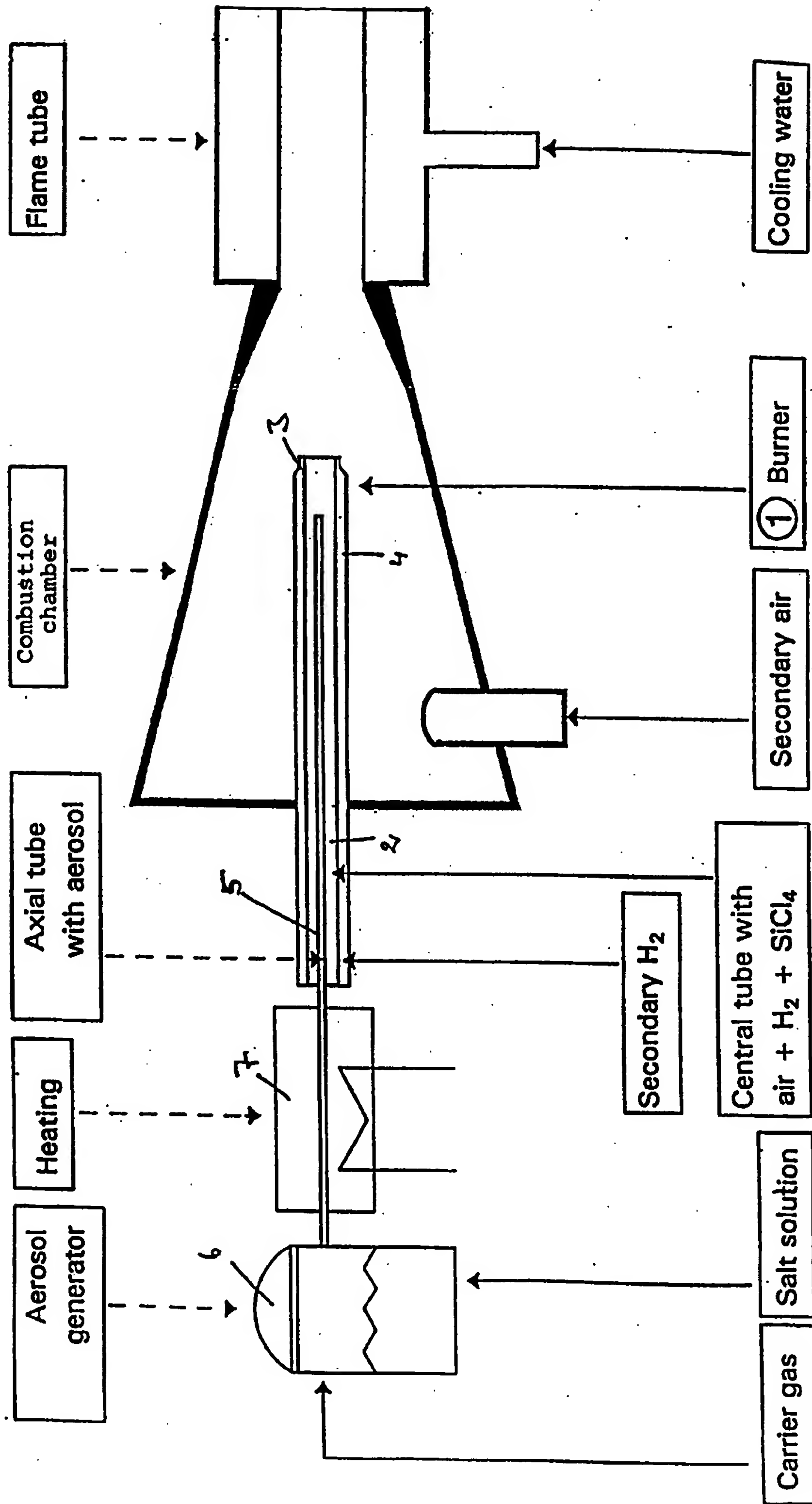
6. A process as defined in Claim 3, 4 or 5, in which the aerosol is produced by ultrasonic nebulisation.

7. The use of doped, pyrogenically-prepared oxides as defined in Claim 1 or 2, as fillers, as support material, as catalytically active substances, as starting materials for preparing dispersions, as polishing materials, as ceramic substrates, in the electronic industry, in the cosmetics industry, as additives in the silicone and rubber industry, to adjust the rheology of liquid systems, for heat-resistant stabilisation purposes, in the lacquer industry and as heat insulation materials.

8. A device for performing the process in accordance with any one of Claims 3 to 6, characterised in that an additional tube for introducing the aerosol into the flame is arranged in a burner of structure for preparing pyrogenic oxides, the tube terminating upstream of the burner nozzle.

9. A device as defined in Claim 8, in which the additional tube for introducing the aerosol into the flame is arranged axially in the burner.

Figure 1:



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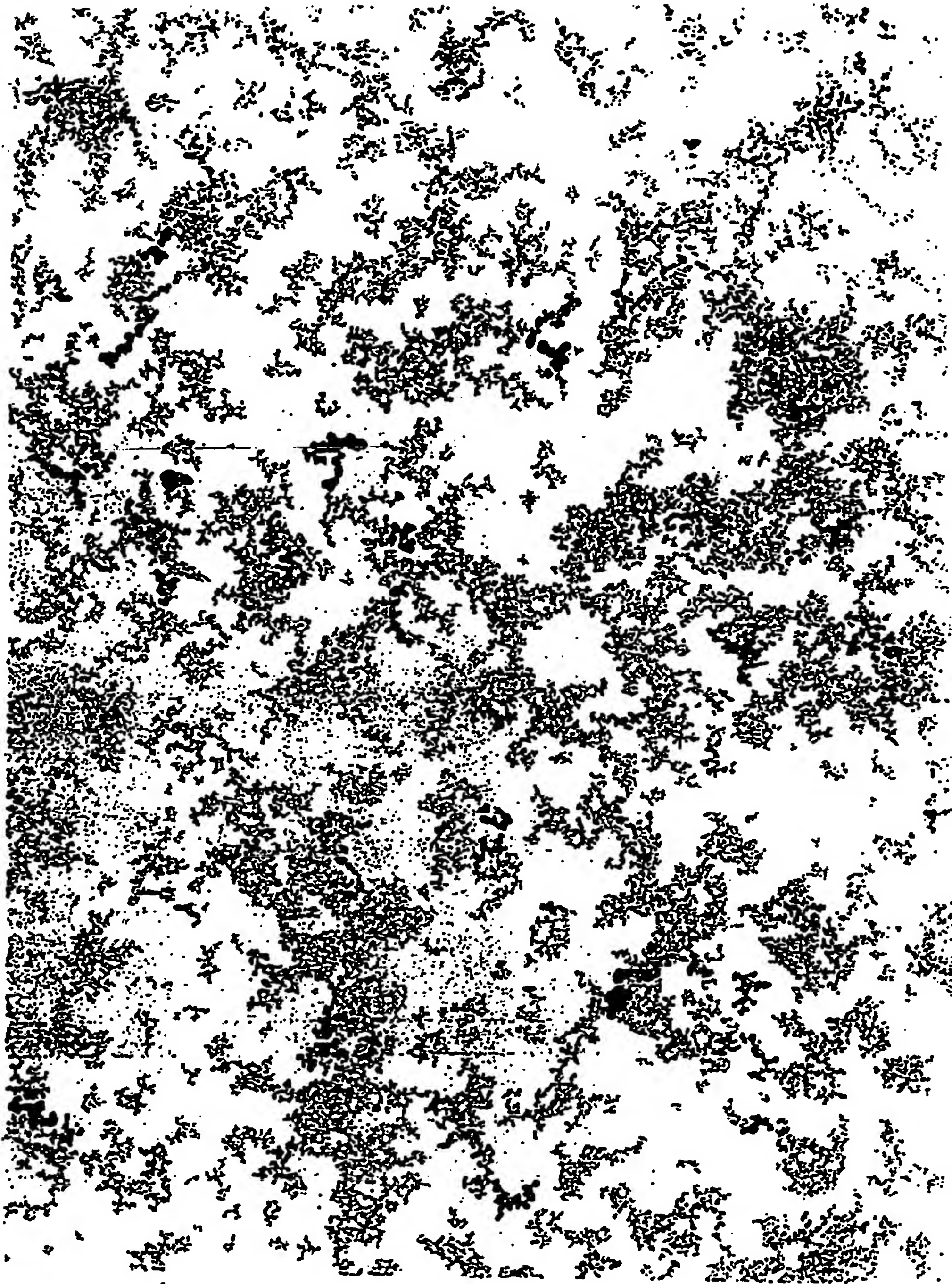


Figure 2

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Figure 3

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